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TECHNICAL REPORT

CASTABLE MATERIALS FOR NEUTRON SHIELDS

By

J. W. McGarvey

and

W. M. Veroeven

UNCLASSIFIED/UNLIMITED

OCO, R and D Branch Project No. 551012285

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--   ELASTIMERIC MATERIALS WHICH ARE KNOWN TO POSSESS INHERENT PHYSICAL
--   PROPERTIES WHICH MAKE THAM ESPECIALLY ATTRACTIVE FOR USE AS THE
--   HYDROGENUOUS COMPONENT OF A RADIOLOGICAL SHIELD. EVALUATION OF ALL
--   THE FACTORS INVOLVED IN THIS PROBLEM MAY REVEAL THEM TO BE THE MOST
--   FEASIBLE MATERIALS FOR THIS PARTICULAR APPLICATION. THE
--   REQUIREMENTS OF THE HYDROGENUOUS MATERIAL ARE SUMMARIZED. THE MAIN
--   CONSTITUENT OF AN ELASTOMERIC NEUTRON SHIELD SHOULD CONSIST OF A
--   BASE POLYMER WHICH EXIBITS A RELATIVELY HIGH HYDROGEN DENSITY. WITH
--   LIQUID POLYMERS SUCH AS THOSE STUDIED, HOWEVER, IT IS POSSIBLE TO
--   SIGNIFICANTLY INCREASE THE HYDROGEN DENSITIES OF THE SYSTEMS BY

-
--   INCORPORATING A HIGH HYDROGEN DENSITY FILLER SUCH AS POLYETHYLENE.
--   THE HYDROGEN DENSITIES OF THE CURATIVES UTILIZED ARE ALSO EXTREMELY
--   IMPORTANT ESPECIALLY WHEN LARGE AMOUNTS ARE REQUIRED. (AUTHOR,
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4 April 1961

OCO, R and D Branch Project
No. 551012285
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Rock Island Arsenal
Rock Island, Illinois

ABSTRACT

Elastomeric materials were investigated as hydrogenous shields for neutron radiation. In general, emphasis was confined to those materials with the highest hydrogen densities which can be cast and cured at room temperature. Various liquid natural, butyl, and polybutadiene formulations were evaluated to determine their curing characteristics and suitabilities as neutron shields. The most promising material consisted of an epoxy resin cured, liquid, carboxy-modified polybutadiene filled with polyethylene.

RECOMMENDATIONS

It is recommended that borated, epoxy resin cured, liquid carboxy modified polybutadiene be considered as a room temperature castable and curable radiological shielding material where fabrication problems are encountered.

It is also recommended that investigations utilizing polymers, fillers and curatives with inherently higher hydrogen densities should be continued.

CASTABLE MATERIALS FOR NEUTRON SHIELDS

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CASTABLE MATERIALS FOR NEUTRON SHIELDS

OBJECT

To develop an elastomeric shielding component which can be cast and cured at room temperature for the purpose of providing personnel with radiological protection.

INTRODUCTION

Definite military advantages could be realized by the employment of personnel provided with radiological shielding if tactical atomic weapons are utilized in future warfare. A suitable radiological shield would attenuate the lethal radiation associated with atomic weapons to tolerable levels, thereby allowing personnel to operate relatively close-in to an explosion.

The dose received by personnel would be primarily due to neutrons and a certain amount of gamma radiation. The gamma radiation results largely from neutrons reacting with elements in the atmosphere, ground, and shielding components.

The attenuation of this lethal radiation must be accomplished in the most efficient manner because of space and weight limitations[1]. It is, therefore, mandatory that the bulky hydrogenous component of such a neutron shield should be composed of a material which exhibits maximum hydrogen density (see Appendix I) and offers minimum fabrication problems.

This investigation was primarily concerned with certain elastomeric materials which are known to possess inherent physical properties which make them especially attractive for use as the hydrogenous component of a radiological shield. In fact, evaluation of all the factors involved in this problem may reveal them to be the most feasible materials for this particular application. In general, the requirements of the hydrogenous material may be summarized as follows:

1. Possess a high hydrogen density.
2. Can be incorporated with an element having a large thermal neutron cross section.
3. Easily processed and fabricated.
4. Possess environmental stability.
5. Composed of nontoxic materials.

The primary purpose of the hydrogenous medium is to slow down neutrons of a definite energy range to allow for their subsequent capture by a suitable element which exhibits a large thermal neutron (see Appendix I) cross section in a reaction other than a (n,γ) process.

In general, capture cross sections are usually larger for thermal neutrons than for high energy neutrons. The most effective neutron shielding can, therefore, be achieved by first decreasing the energy of these particles to a level where a suitable element exhibits a large cross section. Fast neutrons with energies in excess of 1 Mev will be slowed down by elements with fairly high mass numbers in other components of the radiation shield by a process referred to as inelastic scattering (Appendix I). This type of scattering will reduce their energies to about 0.1 Mev where elastic scattering by light elements such as hydrogen is most effective in further reducing their energies to the thermal level. Of all the elements, hydrogen is the best choice for this process because the mass of a hydrogen atom is essentially equal to that of the colliding neutron, thereby allowing for a maximum transfer of energy[2].

The main constituent of an elastomeric neutron shield should, ~~therefore~~, consist of a base polymer which exhibits a relatively high hydrogen density. With liquid polymers such as those studied, however, it is possible to significantly increase the hydrogen densities of the systems by incorporating a high hydrogen density filler such as polyethylene. The hydrogen densities of the curatives utilized are also extremely important especially when large amounts are required. Curatives or polymers containing elements with objectionable (n,γ) interactions should be kept at an absolute minimum. An indication of the undesirability of a particular element can usually be ascertained from a calculation of its macroscopic cross section[3] (Appendix I).

It is also known that the hydrogen and other elements in the radiological shield capture thermal neutrons to a lesser degree in (n,γ) reactions and provisions for attenuating the resulting gamma radiation must be made in other sections of the shield[4,5].

The number of thermal neutrons captured in undesirable (n,γ) reactions can be significantly reduced by incorporating an element such as boron which has a very high thermal neutron cross section to compete for the thermalized neutrons. The reaction whereby a neutron is captured by a boron atom can be expressed as follows[6].

$$_{5}B^{10} + _{0}n^{1} \longrightarrow _{3}Li^{7} + _{2}He^{4} + Q \text{ (Energy)}$$

or

$$_{5}B^{10}(n,\alpha)_{3}Li^{7}$$

The subsequent bombardment of Li^7 with a neutron leads to the formation of two alpha particles together with an electron(7).

Those boron and lithium compounds which were considered as possible fillers or curing agents for the polymers considered in this investigation are given in Appendix II.

The fabrication of the hydrogenous component sometimes introduces engineering problems which are not usually encountered in reactor shielding. A unique solution to the problem of constructing a suitable shielded pod for personnel would be to use a material which could be cast and cured between the inner and outer shells of the pod. Polyethylene which has been utilized as a hydrogenous shield for reactors is not particularly well suited for such an application because of shrinkage problems and the presence of voids when cast(8). The use of polyethylene slabs does not appear practical because of associated fabrication problems.

A list of applicable elastomers and other hydrogenous materials which have been used or considered for shielding applications is given in Appendix III together with their respective hydrogen densities.

This investigation was mainly concerned with obtaining room temperature cures for castable rubbers possessing acceptable hydrogen densities. Gelling agents for saturated hydrocarbons were also investigated, but to a more limited extent, to determine if grease-like materials might offer a practical solution. Those polymers which were evaluated are depolymerized natural and butyl rubbers; liquid carboxy modified polybutadiene; polyisobutylene; and polybutene.

These polymers do not possess the hydrogen density of high density polyethylene, but in some instances they are approximately equal to the low density form which has been used so extensively for shielding applications. It is also known that several of these polymers do not possess inherently good radiation resistance, but anticipated doses fall well below the detectable damage thresholds of even the least radiation resistant polymers.

PROCEDURE

All of the compounds were prepared by mixing the ingredients with a spatula in either 100 X 10 mm petri culture dishes or 50 ml beakers. Specific procedures for the preparation of depolymerized butyl rubber (DPB), butyl latex gels, a hot melt, and greases are given in the following paragraphs.

A solvent free DPB was not commercially available. Initial attempts to obtain a liquid form of butyl rubber were made with a controlled thermal degradation technique. A special apparatus was constructed for uniformly heating a sample of butyl rubber in an evacuated tube and condensing the resulting pyrolysis products. The heater was constructed by wrapping the heating element from a 600 watt cone shaped heater around a 20 X 300 mm test tube and then covering it with refractory cement. With this apparatus, pyrolysis temperatures could be accurately maintained from 250°C - 550°C with a variable transformer.

Butyl rubber was also depolymerized by milling samples with 5 pph rubber of recrystallized dicumyl peroxide, benzoyl peroxide, lead peroxide, a mixture of selected peroxides, and xylyl mercaptan respectively and heating either on a hot plate or in an oven at 170°C for several hours.

Butyl rubber was also depolymerized by gamma radiation from a Co^{60} source. Experiments indicated that a dose of 250 megarads would be sufficient to insure a product which is readily pourable at room temperature.

Butyl latex gels were prepared by mixing commercially available latex and gelling agents. Polyethylene was emulsified with the butyl latex by a wax to water method.

A hot melt was prepared by mixing 100 parts of polyisobutylene with 50 parts of emulsifiable polyethylene and heating to 165°C. The melt was then poured into a petri dish and allowed to cool.

Three aluminum stearate type greases containing 5, 10, and 20 percent aluminum tristearate were prepared by mixing the stearate with a low viscosity polybutene (P-4) at 150°C. The greases were then allowed to cool undisturbed to room temperature. Another grease containing 5 percent aluminum tristearate and 6 percent amorphous boron was prepared in the same manner.

Viscoelastic gels were prepared by melting 1 percent by weight of high density polyethylene and 5 percent by weight of emulsifiable polyethylene with polybutene (P-5) respectively at 200°C.

RESULTS AND DISCUSSION

Depolymerized Rubber

A summary of formulations, degrees of cure, and curing times for depolymerized rubber compounds are listed in Table I. Most of the curing systems evaluated exhibited little or no indication of a cure. Those formulations which resulted in satisfactory cures in most cases lacked sufficiently short cure times to make them of any value for the desired application. A cure time of sufficient length to permit completion of fabrication with optimum handling properties is desired. However, after fabrication a short cure time would be desirable because lengthy cures would permit possible separation of the constituent materials used in the shield. The poor hydrogen density of natural rubber is another of its undesirable properties. As can be seen from the table, the best overall compound was No. 24 which contained activated dithiocarbamate, p-quinonedioxime, and zinc oxide.

Depolymerized Butyl and Butyl Latex

Formulations, cures and curing times for depolymerized butyl and butyl latex can be found in Table II. The butyl latex gels are interesting because they exhibit good hydrogen densities, are castable and gel quickly. The undesirable feature of a butyl latex gel is the entrapped water which comprises 45 percent of the butyl latex. Such a water containing system suffers from dehydration if exposed to the atmosphere and expansion when frozen. For these reasons a butyl latex gel is not considered to be a suitable material for a castable neutron shield.

Depolymerized butyl is attractive due to its high hydrogen density. However, room temperature cures are difficult with liquid butyl due to its low degree of unsaturation. Degrees of cure and curing times at room temperature for depolymerized butyl given in Table II confirm this fact.

Attempts to depolymerize butyl rubber by thermal degradation resulted in low molecular weight products which were unsuitable for this application. Depolymerization with peroxides or various mercaptans has not, at the present time, proven satisfactory.

Radiation depolymerization appears to be the best method of producing a liquid butyl. It is especially attractive in that this product does not contain any undesirable residual elements which may lead to objectionable (n,γ) reactions.

TABLE I

DEPOLYMERIZED NATURAL RUBBER FORMULATIONS

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Depolymerized rubber	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
Polyethylene (powdered)	90							40								
Benzoic acid	1															
Benzothiazyl disulfide		5	1.5	2	2											
Calcium oxide	5					10	10	10	10							
Condensation product of butraldehyde and aniline										5						
Dibenzyl amine											3	3				
Dicumyl peroxide (40% active)													2.5			
Dipentamethylene thiuram tetrasulfide														4	4	4
Diphenyl guanidine		5				10	1	3	3					4	4	4
Dithiocarbamate (activated)			4	8												
Lead dimethyldithiocarbamate		5														
Lead oxide		5								10						
Lead peroxide														20		
Magnesium oxide										5						
2-Mercaptobenzothiazole	2													4	4	4
Refined coal-tar fraction						10										
Sulfur	5	5	1.5	2	2	25	10	30	50		2	2		20		20
Thiocarbanilide					8											
Zinc butyl xanthate											3	3				
Zinc diethyldithiocarbamate											2	2				
Zinc oxide	2									5		4				
Cure*	0	0	1	2	0	1	1	0	1	0	4	3,T	0	1,T	0	0
Time (Weeks)	9	9	9	9	9	9	9	9	9	9	9	9	9	9	9	9

*
0 - No cure
1 - Poor cure
2 - Fair cure
3 - Good cure
4 - Excellent cure

F - Foamed
P - Porous
T - Tacky
G - Gelled

TABLE I (Cont.)

	17	18	19	20	21	22	23	24	25	26	27	28	29	30
Depolymerized rubber	100	100	100	100	100	100	100	100	100	100	100	100	100	100
Polyethylene (powdered)	120	150												
Dipentamethylene thiuram tetrasulfide	2	2	10											
Diphenyl guanidine		2		3										
Dithiocarbamate (activated)				5	5	5	8	4	10					
Lead dimethyldithiocarbamate													5	
Lead oxide					5								5	
Lead peroxide						10				10	20	10		
2-Mercaptobenzothiazole	2	2					3							
Piperidinium pentamethylene dithiocarbamate														5
p-Quinonedioxime										5		5		
Sulfur				5	5	5	3	1	10			5	5	5
Triethanolamine										4				
Trioctyl phosphate		15												
Zinc oxide			1	5		5		5	5	10				5
Cure*	0	0	0	4	0	2,T	0	4	4	3	1,T	1,T	1	4,T
Time (Weeks)	9	9	9	9	9	9	10	1	9	4	10	9	9	9

*
0 – No cure
1 – Poor cure
2 – Fair cure
3 – Good cure
4 – Excellent cure

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P – Porous
T – Tacky
G – Gelled

TABLE II

DEPOLYMERIZED BUTYL AND BUTYL LATEX FORMULATIONS

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Butyl rubber	100	100	100	100	100	100	100	100	100						
Dicumyl peroxide (pure), heated w/butyl - 2 hrs @200°C						10	5	5							
Diphenyl disulfide, heated w/ butyl - 4 hrs @240°C	15	15	15	10	15				10						
Butyl latex										100	100		100	100	100
Butyl latex emulsified w/10% of emulsifiable polyethylene												100			
Polyethylene, emulsifiable											50.5		160	50.5	
Polyethylene powdered			100												
Acrylamide and N,N'-methylene-bisacrylamide mixture										5.05	5.05	5.05		5.05	5.05
Ammonium persulfate										0.252	0.252	0.252		0.252	0.252
Benzothiazyl disulfide		2													
Boron, amorphous														6.07	
B-Dimethylaminopropionitrile										0.202	0.202	0.202		0.202	0.202
Dipentamethylene thiuram tetrasulfide	2		2												
Diphenyl guanidine	2		2												
Dithiocarbamate, activated		2													
Epoxy resin A, diglycidyl ether of bisphenol A				25											
Epoxy resin B, diglycidyl ether of bisphenol A													50		
Epoxy resin, epoxidized polyolefin															22.7
Lead peroxide					40	8	8	10	8						
2-Mercaptobenzothiazole	2		2												
Oxalic acid				2.5									10		2.27
Potassium ferricyanide										0.0076					
p-Quinonedioxime					20	4	4	5	4						
Stearic acid						3	3								
Sulfur	2	2	4			1.5	1.5								
Triethanolamine								4							
Zinc oxide						5	5	10							
Cure*	1	0	0	0	0	1	1	0	0	4,G	4,G	4,G	4,G	4,G	1,G
Time (Weeks)	9	9	9	5	6	3	3	3	5	<1 day	<1 day	<1 day	<1 day	<1 day	2

* 0 - No cure
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Epoxy Resins

Epoxy resin formulations are listed in Table III. These compounds were evaluated in an attempt to optimize both physical properties and chemical composition of an epoxide curing system for a neutron shielding component. As mentioned in the introduction, boron is desirable for thermal neutron capture. It would, therefore, be desirable to incorporate a part or all of the boron needed as an integral part of the shielding material. This would eliminate the risk of having the boron settle out during the curing period as might be the case if elemental boron were used. A boron containing epoxy formulation might be one such approach to this problem.

Of the boron containing compounds evaluated, trimethoxyboroxine and decaborane were found to be most effective as curatives for epoxies of the glycidyl ether of bis(4 hydroxy phenol) dimethyl methane type. It should be noted that decaborane has a tendency to release gas which results in porosity in the cured item. Thus, trimethoxyboroxine, although not as good as decaborane in its cure and cure time, was found to be superior due to its lack of gas formation.

Of the nonboron containing compounds evaluated, oxalic acid was found to be the most effective followed by hexamethylenime as curatives for epoxies of the glycidyl ether of bis (4 hydroxy phenol) dimethylmethane type.

Epoxy Resins and Carboxy-Modified Polybutadiene

These formulations are tabulated in Table IV. Compound No. 8 attains the best cure in the shortest time. This compound, however, possesses poor hydrogen density and contains a fairly high percentage of sulfur and nitrogen. Sulfur is present in the liquid carboxy-modified polybutadiene and nitrogen in the aliphatic polyamine.

Of all compounds evaluated, number twelve is the best potentially castable shield. It exhibits a good hydrogen density (0.120 g/cc) and attains an excellent cure in a relatively short time. Here again, however, sulfur and nitrogen are present. Large concentrations of sulfur and nitrogen are undesirable due to their thermal (n,γ) cross sections.

Liquid Carboxy-Modified Polybutadiene and Boron Compounds

A summary of these formulations with cures and curing times is found in Table V. The primary purpose of the boron compounds in these formulations is as a curative. The chemical composition of decaborane suggests that it would

TABLE III

EPOXY RESIN FORMULATIONS

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Epoxy resin C diglycidyl ether of bis (4 hydroxy phenol) dimethyl methane	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
Boric acid	10														
Decaborane		5													
Hexamethylenimine			5												
Hydroquinone				2	2										
Isopropyl polyborate						10									
Oxalic acid							10								
Pyridine-borane								10							
Pyromellitic dianhydride									10						
Sodium borohydride										10					
Sodium hexylene glycol monoborate											10				
Stannic chloride					2										
Trimethoxyboroxine												10			
Trimethylamine borane													10		
Trimethyl borate														10	
Tri-n-amyl borate															10
Cure*	0	4,P	4	0	1,G	2,T	4	2,T	4	2,F,T	1,G	4	0	0	1,G
Time (Weeks)	5	<1	1	2	4	5	<1	5	<2	5	4	<1	5	5	4

*
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1 - Poor cure
2 - Fair cure
3 - Good cure
4 - Excellent cure

F - Foamed
P - Porous
T - Tacky
G - Gelled

TABLE IV

CARBOXY MODIFIED POLYBUTADIENE - EPOXY RESIN FORMULATIONS

	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Polybutadiene, liquid carboxy modified	100	100	100	100	100	100	100	100	100	100	100	100	100	100
Polybutene, P-1													30	35
Epoxy resin, bis-epoxy-dicyclopentyl ether of ethylene glycol			100											
Epoxy resin A diglycidyl ether of bis (4 hydroxy phenol) dimethyl methane		100				100			25					
Epoxy resin C, diglycidyl ether of bis (4 hydroxy phenol) dimethyl methane	100				50		32.5	65			10	28	28	28
Epoxy resin, epoxidized polyolefin				100						25				
Polyethylene, emulsifiable					100	100					30	100	40	
Polyethylene glycol (liquid)														30
Aliphatic polyamine							17.5	35				12	12	12
Oxalic acid									2.5	2.5	2.5			
Cure*	4,G	4,G	0	2,T,G	4,T	Heated hard wax like solid on cooling	4	4	4	1	3,T	4	4	4
Time (Weeks)	2	2	2	5	5		1	<1	5	5	4	1	1	1

*
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3 - Good cure
4 - Excellent cure

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T - Tacky
G - Gelled

TABLE V

CARBOXY MODIFIED POLYBUTADIENE FORMULATIONS CONTAINING BORON COMPOUNDS

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Polybutadiene, liquid carboxy modified	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
Polyethylene, emulsifiable										50	35				35
Boric acid	10									5	10	10			
Boron oxide												10			
Boron phosphate		10													
Calcium oxide													10	30	
2-Mercaptobenzothiazole										5	10	10			
Isopropyl polyborate			10												
Pyridine-borane				10											
Sodium borohydride					10								2.5	10	10
Sodium hexylene glycol monoborate						10									
Trimethoxy boroxine							10								
Trimethylamine-borane								10							
Trimethyl borate									10						
Zinc oxide										5	10				
Cure*	0	0	1,G	1,G	2,F	2,T	0	0	0	2,T	2,T	0	1	2,F	2,F
Time (Weeks)	5	4	4	4	2	5	4	4	4	4	4	4	5	2	5

*
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1 - Poor cure
2 - Fair cure
3 - Good cure
4 - Excellent cure

F - Foamed
P - Porous
T - Tacky
G - Gelled

TABLE V (Cont.)

	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30
Polybutadiene, liquid carboxy modified	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
Epoxy resin C, diglycidyl ether of bis (4 hydroxy phenol) dimethyl methane	50	25	50	2.8			60	60	60	65	60			30	
Polyethylene, emulsifiable	50	50	50	105				35			35			35	35
Benzoquinone (para)												10			
Decaborane					0.1	10	0.1	1	2.5	10	0.1	2.5	2.5	5	2.5
Lead Peroxide													10		
Trimethoxy boroxine	10	25	50	1.2											10
Cure*	1	1	3	4,P	0	3,F,T	3,G,T	4,P	3,T	3,P,T	4	4,P	4,F	3,P,T	1,P
Time (Weeks)	5	4	<1	<1	5	4	5	5	2	5	5	3	3	2	4

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make an ideal compound for use in a neutron shield. Its use in a liquid carboxy-modified polybutadiene polymer, however, results in gas formation which leads to porosity and foaming in the cured materials ~~(Table V)~~. The present high cost of decaborane and its toxic qualities also tend to prohibit its use for this application.

Liquid Carboxy-Modified Polybutadiene and Accelerators

A summary of these formulations is found in Table VI. Extensive efforts were made early in the program to find an accelerator type curing system for the liquid carboxy-modified polybutadiene. This was a logical approach due to the extensive use of accelerators in the rubber field and literature references listing room temperature cures with them.

It has become apparent, however, that the amounts of accelerators and activators required for a satisfactory room temperature cure would excessively reduce the hydrogen density of the final shield material. In most cases, accelerators contain fairly high percentages of sulfur and show little promise unless an ultra-accelerator can be found that requires only small quantities to effect room temperature cures.

Polyisobutylene and Polybutene Formulations

The hot melt consisting of 100 parts polyisobutylene and 50 parts emulsifiable polyethylene offers an attractive possibility because of its inherently good hydrogen density, the absence of objectionable elements and the compatibility of the components. When cooled, the material is a very viscous semi-solid. Normally, the softest grade of polyisobutylene does not become sufficiently fluid to be pumped even when heated to 150°C. When emulsifiable polyethylene is blended with it, however, the material is readily pourable at elevated temperatures.

Satisfactory greases composed of 5 and 10 percent aluminum tristearate and 95 and 90 percent polybutene P-4 respectively were prepared. Attempts to prepare a grease with 20 percent aluminum tristearate resulted in a hard brittle soap-like material. The consistency of a grease containing 5 percent soap and 6 percent amorphous boron was very nearly the same as the one prepared without the boron.

The viscoelastic gels prepared with either high density polyethylene or emulsifiable polyethylene and polybutene P-5 were not very strong and are probably not applicable for the intended application.

TABLE VI

CARBOXY MODIFIED POLYBUTADIENE - ACCELERATOR FORMULATIONS

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Polybutadiene, liquid carboxy modified	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
Polyethylene, emulsifiable	50	25	15	50									40	40	50
2 (allylthio) benzothiazole										10	10	5	2	5	2.5
Dibenzyl amine	1.5							1.5	1.5						
Dipentamethylene thiuram tetrasulfide										10	10				2.5
Dithiocarbamate (activated)		5	10	10	5										
Piperidinium pentamethylene dithiocarbamate							5						5	5	
Reaction product of carbon bisulfide and methylene dipiperidine						10									
Sulfur	3	5	5	5	5	5	3	3	3	5	5	2.5	2	2.5	2.5
Tetramethyl thiuram disulfide												5	2		
Zinc butyl xanthate	3							3	3						
Zinc oxide	10	5	5	5	5	5	5	10	10	10		10	4	5	7.5
Cure*	4	4	4	4	4	4	3	4	4	3,T	0	2,T	2,T	1	3,T
Time (Weeks)	6	<2 Heated @100°C then poured	2 Heated @100°C for 1/2 hr. and poured	<2 Heated to 100°C and poured	2 Heated to 100°C and poured	3	7	<2	<2	5	5	5	5	5	5

*
0 - No cure
1 - Poor cure
2 - Fair cure
3 - Good cure
4 - Excellent cure

F - Foamed
P - Porous
T - Tacky
G - Gelled

TABLE VI (Cont.)

	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31
Polybutadiene, liquid carboxy modified	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
Polyethylene, emulsifiable									100						50	50
2(allylthio) benzothiazole													5			
Benzoquinone – para		10														
Carbon disulfide												5				
Dibenzyl amine												5		1.5	1.5	
Dicumyl peroxide (40% active)					5	10										
Dipentamethylene thiuram tetrasulfide										10	10					5
Dithiocarbamate (activated)	10					10	5	5	5							
Lead peroxide			10				5									
2-Mercaptobenzothiazole										10	10	5				5
Piperidinium pentamethylene dithiocarbamate													5			
Reaction product of carbon bisulfite and methylene dipiperidine				10												
Sulfur							5	5	5			5	5	3	3	
Tetraethyl thiuram disulfide													5			
Zinc butyl xanthate												5		3	3	
Zinc oxide								5	5	10		5	10 Heated 100°C	10	10	5
Cure*	0	0	0	0	0	1	1,G	3,T	Heated to 110°C. Hard wax like solid on cooling	3,T	0	3,T	4	4	4	4
Time (Weeks)	3	3	2	2	<2	4	<2	1		<2	5	1	<1	<2	<5	5

*
0 – No cure
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TABLE VI (Cont.)

	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48
Polybutadiene, liquid carboxy modified	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
Polyethylene emulsifiable				35	35			40	40	40	30	30	30		30		50
2(allylthio) benzothiazole		5	5		5											5	5
Benzothiazole										2.5							
Dipentamethylene thiuram tetrasulfide			5	5													5
Glycoldimercaptoacetate							1.8										
Lead oxide						5											
Lead peroxide	8													8			
2-Mercaptobenzothiazole				5		5		2.5	2		5	5	2.5		5		
Piperidinium pentamethylene dithiocarbamate		5			5							5	5		5	5	
Quinonedioxime - para	4													4			
Selenium diethyldithiocarbamate						5											
Stearic acid							1										
Sulfur		2.5			5				1				2.5		5	2.5	
Tetraethylthiuram disulfide					5												
Tetramethylthiuram disulfide								2.5	2	2.5	5	5	2.5		5		
Zinc oxide	5	5	10	5	10		3	5	5	5	10	10	5	5	10	5	10
Cure*	3,T	3,T	3,T	3,T	4	0	0	2,T	2,T	2,T	3,T	2,T	2,T	4	4	4,T	2,T
Time (Weeks)	3	5	5	5	5	6	4	5	5	4	4	4	5	6	5	9	6

*
0 - No cure
1 - Poor cure
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3 - Good cure
4 - Excellent cure

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CONCLUSIONS

An epoxide curing system for a liquid carboxy-modified polybutadiene polymer containing polyethylene (Composition No. 12, Table IV) has been found the most satisfactory system for use as a castable neutron shield. The theoretical hydrogen density for this system is 0.120 gms/cc*. Possibilities exist for increasing the hydrogen density to 0.125 gms/cc by using high hydrogen density polyethylene and a poly(allyl glycidyl ether) type epoxy which has greater hydrogen density than the diglycidyl ether of bis(4 hydroxyphenol) dimethyl methane epoxy resin. The best commercially available castable shield is reported to have a hydrogen density of 0.118 gms/cc.

Studies and investigations involving depolymerized natural rubber and butyl latex indicate them to be unsuitable for use in a castable neutron shield. It is believed that a liquid butyl rubber shows promise if a suitable room temperature cure can be found.

It has also been found that most shielding formulations incorporating fairly large amounts of polyethylene powder contain entrapped air which results in porosity of the end products. Attempts at removing this entrapped gas under vacuum, after mixing, have been unsuccessful. It is believed, therefore, that mixing under vacuum will be necessary in order to alleviate the problem of entrapped air.

Future efforts should be directed to improving the best formulations available at the present time. It is also felt that a more thorough investigation of the possibilities offered by depolymerized butyl polymers should be carried out.

*Calculated using manufacturers value for percentage hydrogen in liquid carboxy-modified polybutadiene.
Hydrogen density for the polyamide was obtained using percentage hydrogen as obtained by a private laboratory.

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APPENDIX I

Definitions

Barn - An effective cross section of $10^{-24} cm^2$.

Elastic scattering - A collision in which kinetic energy is transferred from the projectile to the target nucleus but the latter is not raised to an excited state.

Hydrogen density - Expressed as mass of hydrogen per unit volume of the parent compound, and is obtained by multiplying the percentage of hydrogen by the density of the compound.

Inelastic scattering - A collision in which the kinetic energy lost by the projectile has been converted into excitation (or potential) energy in the target nucleus.

Macroscopic cross section (Σ) - The product of the microscopic cross section (σ) and the number of nuclei/ cm^3 (N). Or simply: $\Sigma = N\sigma \ cm^{-1}$.

Megarad - A dose of 10^8 ergs of absorbed energy per gram of material.

Microscopic cross section (σ) - The circular area which a nucleus must have if each collision within this cross section would produce a transformation.

Thermal neutrons - Neutrons which are in thermal equilibrium with the atoms or molecules of the surrounding medium. At 25°C thermal neutrons have an average energy of 0.026 ev and a speed of 2.2×10^5 cm/sec.

Total cross section - The sum of the scattering and absorption cross sections for a given nuclide. The former is the sum of cross sections for elastic and inelastic scattering and the latter is the total cross section for all processes in which a neutron is captured and another particle (or particles) emitted.

APPENDIX II

Boron and Lithium Compounds

Boron and Lithium Compounds

Name and Formula	Mol. Wt.	Density g/ml	Sol. g per 100 ml H_2O	% B	% H	H Density g/ml
Boric acid, H_3BO_3	61.84	1.435	5.15^{21} 39.1^{100}	17.49	4.85	0.069
Boric acid, fused, B_2O_3	69.64	1.805	Cold sl. s. Hot s.	31.07	0	0
Boron, Amorphous, B	10.82	2.34	i	100	0	0
Boron carbide, B_4C	55.29	2.50	i	78.28	0	0
Boron fluoride ethyl ether $BF_3O\ (C_2H_5)_2$	141.94					
Boron nitride, BN	24.83	2.20	i	43.58	0	0
Boron phosphate	105.80	-	-	10.22	0	0
Decaborane, $B_{10}H_{14}$	122.31	0.94	s. Inert hydrocarbons	88.4	11.54	0.108
Isopropyl polyborate $[(CH_3)_2CHO]_6\ B_8O_9$	585.09	-	"	14.79	7.23	-
Lithium borohydride, $LiBH_4$	21.79	0.68	d.	49.65	18.49	0.126
Lithium tetraborate $Li_2B_4O_7 \cdot 5H_2O$	259.24	-	v.s.	16.69	3.9	-
Pentaborane	63.17	0.630	-	85.64	14.36	0.09
Pyridine-borane $C_2H_5N{:}BH_3$	92.95	0.92	-	11.64	8.67	0.080
Sodium borohydride Na BH_4	37.85	1.07	d.	28.58	10.65	0.114
Sodium hexalene glycol mono-borate $CH_2(CH_3)_2\ C(CH_3)CHOBONa$	165.98	0.25	s non-polar sol.	6.5	7.3	0.018

Boron and Lithium Compounds (Cont.)

Name and Formula	Mol. Wt.	Density g/ml	Sol. g per 100 ml H_2O	% B	% H	H Density g/ml
Triallyl borate $(CH_2 = CHCH_2O)_3B$	182.03	0.916	Hydrolyzes	5.94	8.31	0.076
Tridecyl borate $(C_{10}H_{21}O)_3B$	481.8	0.858	-	2.24	13.18	0.113
Trimethyoxy boroxine $(CH_3OBO)_3$	173.56	1.22	-	18.59	5.22	0.064
Trimethylamine borane $(CH_3)_3N:BH_3$	72.96	-	d	14.8	16.47	-
Trimethyl borate $(CH_3O)_3B$	103.92	0.9205	s. eth.	10.41	8.72	0.080
Tri-n-amyl borate $[CH_3(CH_2)_4O]_3B$	272.24	0.872	-	3.97	12.22	0.107
Tri-n-butyl borate $[CH_3(CH_2)_3O]_3B$	230.16	0.864^{0}	-	4.61	11.82	0.102
Tri-o-cresyl borate $(C_7H_7O)_3B$	332.2	1.08^{22}	-	3.26	6.37	0.069

APPENDIX III

Physical Constants of Various Shielding Materials

Boron and Lithium Compounds

Material	Formula	Sp. g	H Density
Aromatic hydrocarbons		0.85 1.05	0.078 0.113
Butyl latex, 55% solids	$(C_{245}H_{488})_n + H_2O$	0.96	0.123
Butyl rubber	$(C_{245}H_{488})_n$	0.92	0.130
Castable rubber-base material	-	0.94	0.118
Epoxy resin, A,B,C, diglycidyl ether of bis (4 hydroxy phenol) dimethyl methane	$(C_{21}H_{24}O_4)_n$	1.16 1.13 1.18	0.083
Epoxy resin, epoxidized polyolefin	$(C_{22}H_{34}O_5)_n$	1.010	0.091
Epoxy resin, polyallyl glycidyl ether	$(C_{24}H_{40}O_8)_n$	1.128	0.099
Fuel Oil	-	0.89	0.106
Gasoline	C_7H_{16} ave.	0.72	0.115
Lucite	$(C_5H_8O_2)_n$	1.2	0.096
Natural rubber	$(C_5H_8)_n$	0.92	0.109
Paraffins	C_nH_{2n+2}	0.87 0.91	0.129 0.133
Polybutadiene, hydrogenated	-		
Polybutadiene, liquid	-	0.908	0.095
Polybutadiene, liquid carboxy modified	-	0.090	0.123
Polybutenes		0.83 0.919	0.119 0.131
Polyethylene	$(CH_2)_n$	0.92 0.96	0.131 0.137
Polyisobutylene	-	0.92	0.131
Polypropylene	$(C_3H_6)_n$	0.89 0.92	0.129
Polystyrene	$(C_8H_9)_n$	1.05	0.089
Water	H_2O	1.00	0.111
Grease	Polybutene + 5% Aluminum stearate		0.119 0.130

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CASTABLE MATERIALS FOR NEUTRON SHIELDS

by J. W. McGarvey and W. M. Veroeven

RIA Lab. Rep. 61-1344, 4 Apr 61, 30 p. incl. illus. tables, (Project No. 551012285, DA Project No. 548-03-003) Unclassified report.

Elastomeric materials were investigated as hydrogenous shields for neutron radiation. In general, emphasis was confined to those materials with the highest hydrogen densities which can be cast and cured at room temperature. Various liquid natural, butyl, and polybutadiene formulations were evaluated to determine their curing characteristics and suitabilities as neutron shields. The most promising material consisted of an epoxy resin cured, liquid, carboxy-modified polybutadiene filled with polyethylene.

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CODE SHEET

Chemical Name or Description	Trade Name or Common Name	Source
Depolymerized rubber	DPR	DPR, Inc.
Polyethylene (powdered)	Alathon F	E.I. du Pont de Nemours & Company
Condensation product of butraldehyde & aniline	Accelerator 808	" "
Dicumyl peroxide (40% active)	Di-Cup 40C	Hercules Powder Co.
Dithiocarbamate, activated	Butyl 8	R.T. Vanderbilt Co., Inc.
Refined coal-tar fraction	Bardol	Allied Chem. & Dye Corp.
Butyl rubber	Enjay 325	Enjay Co., Inc.
Butyl latex	Enjay Butyl Latex 80-21	" "
Polyethylene, emulsifiable	Polyethylene 617-A	Allied Chem. & Dye Corp.
Acrylamid and N,N'-methylenebisacrylamid mixture	Cyanogum 41	Am. Cyanamide Co.
Epoxy resin A, diglycidyl ether of bis(4 hydroxyphenol) dimethyl methane	Gen Epoxy 175	Gen. Mills, Inc.
Epoxy resin B, diglycidyl ether of bis(4 hydroxy phenol) dimethyl methane	Gen Epoxy 180	" "
Epoxy resin, epoxidized polyolefin	Oxiron 2000	Food Mach. & Chem. Corp.
Epoxy resin, polyallyl glycidyl ether	Shell X-101	Shell Chem. Co.

CODE SHEET (Cont.)

Chemical Name or Description	Trade Name or Common Name	Source
Epoxy resin C, diglycidyl ether of bis(4 hydroxy phenol) dimethyl methane	Gen Epoxy 190	Gen Mills, Inc.
Epoxy resin, bis-epoxy-dicyclopentyl ether of ethylene glycol	Diepoxide AG-13E	Rohm & Haas Co.
Polybutadiene, liquid carboxy modified	Hycar 2000 X 131	B.F. Goodrich Chem. Co.
Polybutene, P-1	Polybutene 24	Oronite Chem. Co.
Polybutene, P-4	Polybutene L-100	Amoco Chemical Corp.
Polybutene, P-5	Polybutene H-100	" "
Aliphatic polyamine	Genamid 250	Gen. Mills, Inc.
Reaction product of carbon bisulfide and methylene dipiperidine	R-2 Crystals	Monsanto Chem. Co.
A mixture of selected peroxides	Vorox	R.T. Vanderbilt Co.,Inc.
Polyisobutylene	Vistenex LM-MS	Enjay Chem. Co.
High density polyethylene	Morlex 6000 Type 9	Phillips Chem. Co.

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